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THE EFFECT OF ALLOYING ON MECHANICAL PROPERTIES OF ADVANCED HIGH STRENGTH STEELS

WPŁYW DODATKÓW STOPOWYCH NA WŁAŚCIWOŚCI MECHANICZNE ZAAWANSOWANYCH STALI O DUŻEJ WYTRZYMAŁOŚCI

Quenching and partitioning process with incorporated incremental deformation was optimized for six high strength steels with various contents of carbon (0.4-0.6%), manganese (0.6-1.2), silicon (2-2.6%) and chromium (0.8-1.3%). The optimization was gradually done for each steel with respect to the final microstructures and properties. The effect of cooling rate, quenching and partitioning temperature on microstructure development was further investigated. Interesting combinations of mechanical properties were obtained, with tensile strength in the region of 1600-2400 MPa and ductility of 6-20%.

Keywords: quenching and partitioning process, retained austenite, martensite, incremental deformation

Przeprowadzono optymalizację procesu hartowania i partycjonowania węgla (*Quenching and Partitioning – Q&P*) w połączeniu ze stopniowymi przyrostami odkształcenia dla sześciu wysokowytrzymałych stali o różnej zawartości węgla (0,4-0,6%) manganu (0,6-1,2%), krzemu (2-2,6%) oraz chromu (0,8-1,3%). Optymalizację prowadzono dla każdej stali stopniowo pod kątem końcowej mikrostruktury i właściwości. Następnie przeprowadzono badania wpływ szybkości chłodzenia oraz temperatury hartowania i partycjonowania na rozwój mikrostruktury. W rezultacie otrzymano interesującą kombinację właściwości mechanicznych stali o wytrzymałości na rozciąganie w zakresie 1600-2400 MPa oraz ciągliwości w zakresie 6-20%.

1. Introduction

New innovative strategies of heat and thermo-mechanical treatment fulfil the need for cost efficient low alloyed steels with high ultimate strength and good ductility. These interesting combinations of mechanical properties can be obtained by controlled microstructure development. New kinds of steels which are utilizing the positive effect of retained austenite on strength to ductility balance of low alloyed steels have been recently developed [1]. This effect is successfully used also for martensitic steels produced by quenching and partitioning (Q-P) process [2].

Even further enhancement of mechanical properties can be achieved when the Q-P process is combined with intensive incremental deformation carried out in the austenite region to obtain finer martensitic matrix [3]. The amount of applied deformation might also influence morphology and stability of the retained austenite [4]. This combination of heat treatment and controlled deformation has an important practical impact, as incremental deformation is a fundamental principle of several processing methods used in industrial practice. To obtain a good combination of mechanical properties it is however necessary to optimize several parameters of Q-P process [5]. Among the most important ones are austenisation temperature, cooling rate, quenching temperature and partitioning hold. The typical chemical composition of steels used for Q-P process is around 0.4%C, 1-2%Si, 1-2Mn, with optional further alloying with chromium, nickel, molybdenum or microalloying with niobium [6, 7]. The first practical applications of these steels are in hot stamping of parts for automotive industry [8].

2. Experimental Program

The aim of this work was to investigate the influence of various alloying elements on Q-P process parameters, final microstructure and properties. Among the most important parameters for Q-P process, which will be treated in this work, are the cooling rate from austenisation temperature to the quenching temperature, quenching temperature and partitioning temperature and hold. A thermo-mechanical simulator was used for sample processing, as it applies precisely controlled and monitored thermal and deformation regimes, which might include rapid incremental deformations. These abilities allow precise temperature and deformation parameters to be set up, similar to the real process of technology or material development. The samples for simulator have cylindrical active part with the diameter of 8 mm and the length of 16 mm. The overall length of the sample including threads at both ends is 76 mm. This geometry of the sample was optimized to achieve homogeneous thermal field distribution in active part of the sample.

Six high strength steels with slightly different chemical compositions were used in this work. Carbon content var-

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ied from 0.4 to 0.6% and various contents of manganese (0.6-1.2%), silicon (2-2.6%) and chromium (0-1.3%) were chosen to investigate the influence of these main alloying elements on the final microstructures and mechanical properties (Tab. 1). Different amounts of alloying elements are also responsible for slightly different temperature intervals of martensitic transformation. Thermal parameters of Q-P process therefore had to be optimized with respect to these temperatures.

Steel	C	Si	Mn	Cr	Ms	\mathbf{M}_{f}
0.4C-2Si-0.6Mn-1.3Cr	0.4	2.0	0.6	1.3	298	178
0.4C-2.6Si-0.6Mn-1.3Cr	0.4	2.6	0.6	1.3	309	189
0.4C6Si-1.2Mn-1.3Cr	0.4	2.6	1.2	1.3	276	153
0.4C-2Si-0.6Mn	0.4	2.0	0.6	_	320	202
0.6C-2Si-0.6Mn-0.8Cr	0.6	2.0	0.6	0.8	263	138
0.6C-2Si-0.6Mn-1.3Cr	0.6	2.0	0.6	1.3	252	126

Chemical composition (wt %), M_s and M_f temperature (°C)

TABLE 1

The final microstructures were analysed by laser scanning confocal microscopy, scanning electron microscopy and transmission electron microscopy. Volume fractions of retained austenite were measured by X-ray diffraction phase analysis, using AXS D8 Discover X-ray analyser with Co K α radiation.

Mechanical properties in terms of tensile strength and ductility were determined by tensile test of flat mini-specimens with 5 mm length and 1.2×2 mm cross section of the active part of the specimen. Two samples per process condition were subjected to tensile testing and the average value was reported.

2.1. Thermo-mechanical processing

Thermo-mechanical processing strategies with different cooling rates were designed for each steel (Tab. 2, Fig. 1). On the basis of previous optimization experiments performed on 0.4C-2Si-0.6Mn steel [4], thermo-mechanical processing with 20 incremental deformation steps and incorporated Q-P treatment was chosen for all the steels. The heating temperatures were designed with respect to the highest cost efficiency of the processing. Therefore the lowest temperatures which ensured full austenisation of the steels were used. The most suitable temperature for all chromium steels turned out to be 950°C. Only the chromium-less steel needed higher heating temperature of 1000°C. Incremental deformation was always applied during the cooling from austenisation temperature in the form of 20 subsequent tension and compression deformation steps, with the total logarithmic deformation Φ equal to 5. Finishing hot-working temperature of 720°C was used for all processing with heating temperature 950°C. To ensure that all deformation is carried out in austenite region, higher finishing temperature of 850°C was used for the processing of chromium-less steel with 1000°C heating temperature. As the cooling rate is one of the most important parameters of each thermo-mechanical processing, four different average cooling rates 50, 30, 20 and 7°C/s were applied to cool the steels after incremental deformation to quenching temperature. Quenching temperature of 200°C was used for the reference treatment, based on previous results obtained at 42SiCr steel [3].

The most convenient cooling rates for the four steels with higher chromium content (1.3%Cr) turned out to be 20°C/s. Isothermal hold of 250°C with holding time 600s (Fig. 1) was again chosen on the basis of previous experiments [3-5].

Further optimization of quenching and partitioning temperatures was carried out for each steel and is described in more detail elsewhere [9,10].

The steel without chromium needed not only higher heating temperature of 1000°C but also significantly higher cooling rates to avoid pearlite and free ferrite formation. Cooling rates 30°C/s and 50°C/s were therefore applied to achieve martensitic microstructure.

On the other hand, the steel with higher chromium (1.3%) and higher carbon (0.6%) content required lower quenching and partitioning temperatures and shorter hold $(175^{\circ}C/10s-200^{\circ}C/100s)$ to obtain the best mechanical properties.



Fig. 1. Parameters of Q-P process with integrated incremental deformation

TABLE 2 Thermo-mechanical treatment parameters, mechanical properties, retained austenite volume

Steel	Processing	Cooling rate [°C/s]	RA [%]	R _m [MPa]	A _{5mm} [%]					
0.4C-2Si- 0.6Mn-	950°C/100s – 200°C/10s – 250°C/600s	20	17	2096	12					
1.3Cr		7	14	1879	10					
0.4C-2.6Si- 0.6Mn-		20	13	1965	17					
1.3Cr		7	23	1791	14					
0.4C6Si- 1.2Mn-		20	14	2118	14					
1.3Cr		7	15	1938	17					
0.6C-2Si- 0.6Mn-		20	22	2100	19					
0.8Cr		7	28	1877	16					
0.6C-2Si- 0.6Mn-		20	16	2168	8					
1.3Cr	950°C/100s – 175°C/10s – 200°C/100s	20	14	2438	14					
0.4C-2Si	950°C/100s – 200°C/10s – 250°C/600s	20	20	1959	15					
	1000°C/100s- 200°C/10s-	30	4	1367	7					
	250°C/600s	50	4	1300	16					

3. Results and discussion

3.1. Effect of cooling rate on microstructure and properties

The processing with a cooling rate of 20°C/s resulted in a typical Q-P microstructure consisting of a mixture of martensitic matrix with lower bainite and 13-17% of retained austenite for the steels 0.4C-2Si-0.6Mn-1.3Cr. 0.4C-2.6Si-0.6Mn-1.3Cr. 0.4C-2.6Si-1.2Mn-1.3Cr and 0.6C-2Si-0.6Mn-0.8Cr (Fig.2). The austenite mainly forms thin films at the martensitic laths boundaries (Fig.3). For middle carbon (0,4%) and high chromium (1,3%) steels this microstructure reached the highest ultimate strength around 1965MPa - 2118 MPa and ductility of 14-17%. Slower cooling by 7°C/s already enabled ferritic and pearlitic transformations to occur in both 0.4C-2Si-0.6Mn and 0.4C-2.6Si-0.6Mn steel and therefore small islands of pearlite and free ferrite were found in the final microstructures. The amount and size of ferrite grains and pearlite areas were higher for high silicon 0.4C-2.6Si-0.6Mn steel, which was also reflected by its lower strength.



Fig. 2. 0.4C-2Si-0.6Mn steel, $950^{\circ}C - 200^{\circ}C - 250^{\circ}C/600$ s, cooling rate $20^{\circ}C/s$, martensitic microstructure with lower bainite, SEM



Fig. 3. 0.4C-2Si-0.6Mn steel, 950° C – 200° C – 250° C/600 s, cooling rate 20° C/s, dark field image of retained austenite (white) in martensitic microstructure, TEM

At a lower cooling rate, the 0.4C-2.6Si-1.2Mn steel alloyed with higher contents of silicon and manganese showed different behaviour from the low silicon middle carbon steels. The main difference was the absence of grown pearlite colonies in the final microstructure even after the slow cooling by 7°C/s (Fig. 4). Another interesting feature is that this cooling rate did not result in any significant drop of mechanical properties, as the ultimate strength was still nearly 2000 MPa.

For the steel with higher carbon and high chromium content 0.6C-2Si-0.6Mn-1.3Cr, the application of cooling rate 20°C/s resulted in mostly martensitic microstructure with small amounts of lower bainite and 16% of retained austenite (Fig. 5). Even though the steel achieved high strength above 2100 MPa, the ductility was low, reaching only 8%. To improve strength to ductility balance, various combinations of quenching temperature and partitioning hold were tested. The most suitable turned out to be quenching to 175°C followed by 100s hold at 200°C, which resulted in the highest strength of 2438 MPa with good ductility of 14%.

The final microstructure of high carbon low chromium steel 0.6C-2Si-0.6Mn-0.8Cr after slow cooling consisted of a mixture of bainite and free ferrite. The faster cooling by 20°C/s produced predominantly martensitic microstructure with 22% of retained austenite. Only a few areas of lower bainite or laths of bainitic ferrite were sporadically found in this microstructure (Fig. 6), which possessed tensile strength of 2100 MPa combined with excellent ductility of 19%. The highest amount of retained austenite was also detected in this steel after 20°C/s cooling.

Due to the lack of chromium, martensite start temperature of 0.4C-2Si-0.6Mn steel increased to 320°C and pearlite and ferrite transformations were also shifted to the shorter times [9]. High cooling rates were therefore required for the processing of this steel. First, reference treatments with 20°C/s, 30°C/s and 50°C/s cooling rates from 950°C were applied to the steel. The final microstructures consisted of ferrite, bainite and martensite. To obtain predominantly martensitic microstructure, higher cooling rates of 30°C and 50°C/s were tested in combination with higher austenisation temperature of 1000°C. Desired martensitic microstructure (Fig. 7) was finally obtained after cooling by 50°C/s, however upper bainite was still present at original austenite boundaries, the amount of retained austenite was very low and the ductility reached 16%. Tensile strength of these microstructures was around 1300 MPa, which was even below the strength of complex bainitic-ferritic-martensitic microstructure (1959 MPa) obtained after slower cooling of the same steel.



Fig. 4. 0.4C-2.6Si-1.2Mn steel, $950^{\circ}C - 200^{\circ}C - 250^{\circ}C/600s$, cooling rate 7°C/s, final microstructure without pearlite and large volumes of free ferrite, laser scanning confocal microscopy



Fig. 5. 0.6C-2Si-0.6Mn-1.3Cr, $950^{\circ}C - 200^{\circ}C - 250^{\circ}C/600s$ cooling rate $20^{\circ}C/s$, martensitic microstructure with small amount of lower bainite, SEM



Fig. 6. 0.6C-2Si-0.6Mn-0.8Cr , $950^{\circ}C - 200^{\circ}C - 250^{\circ}C/600s$, cooling rate $20^{\circ}C/s$, martensitic microstructure with two ferrite laths and small amount of lower bainite, SEM



Fig. 7. 0.4C-2Si-0.6Mn, 1000° C/ $100s - 200^{\circ}$ C - 250° C/600s, cooling rate 50° C/s, martensitic microstructure with bainite sporadically formed at original austenite boundaries, SEM

Even though the original theory of Q-P process spoke about martensitic final microstructure with controlled fraction of retained austenite [2], the results obtained since than by many researchers [5-7, 10] proved, that various amounts of upper bainite are usually part of the final microstructures as well. This bainite could even in silicon-based steels contain ε carbides [11], as it was demonstrated [7] that silicon is not so efficient in inhibiting epsilon carbide precipitation as it is in the case of cementite. The final microstructures obtained in this work consisting mostly of the mixture of martensite, upper bainite and retained austenite are therefore not exceptional and are in good agreement with other findings published at similar steels after Q-P process [5-7, 10].

4. Conclusions

The optimization of processing parameters enabled six steels with various contents of carbon (0.4-0.6%), chromium (0-1.3%), manganese (0.6-1.2%) and silicon (2-2.6%) to obtain a typical Q-P microstructure consisting of the mixture of martensitic matrix with lower bainite and 6-22% of retained austenite. The ultimate strengths of 1300-2243 MPa were obtained with ductility A_{5mm} of 7-21%.

The steel with the highest silicon (2.6%) and manganese (1.2%) contents showed lower sensitivity to the cooling rate effect, avoiding the formation of ferrite-pearlite areas in the final microstructures even after slow cooling by 7°C/s.

The chromium-less steel was found unsuitable for Q-P processing, due to its low quenchability and low strength of obtained martensitic microstructure.

The combination of high carbon (0.6%) and high chromium (1.3%) contents on the other hand resulted in the highest strength of 2438 MPa with relatively high 14% ductility. However, lower quenching temperature of 175°C, followed by short 100s hold at 200°C had to be applied to achieve these properties.

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REFERENCES

- S. W i e w i ó r o w s k a, Archives of Metallurgy and Materials 58, 573-578 (2013).
- [2] D.V. E d m o n d s, et al. Materials Science and Engineering A 438-440, 25-34 (2006).
- [3] B. M a š e k, et al. Journal of Iron and Steel Research International **18**, 730-734 (2011).
- [4] L. Kučerová, et al. in, Annals of DAAAM for 2009, DAAAM International Vienna, 1035-1036, Vienna (2009).
- [5] B. Mašek, et al. Materials Science Forum **654-656**, 94-97 (2010).
- [6] B. Paravicini, et al., Materials Science and Engineering: A 559, 486-495 (2013).
- [7] S.S. Nayak, et al., Materials Science and Engineering: A 498, 442-456 (2008).
- [8] L. H e p i n g, et al., Scripta Materialia 64, 749-752 (2011).
- [9] H. Jirková, et al., Material Science Forum 706-709, 2734 (2012).
 [10] D. Aišman, et al., in: Annals of DAAAM for 2008, DAAAM
- International Vienna, 1035-1036, Vienna (2008).
- [11] H.Y. L i, et al., Materials Science and Engineering A 527, 6255-6259 (2010).